

As originally filed

Dye preparations

5

The present invention relates to dye preparations and their use, especially for ink jet printing.

10 DE-A 197 52 333 discloses dye formulations which are used for ink jet printing. They contain polymeric dispersants which are specific arylsulfonic acid-formaldehyde condensates as known from US 5,186,846.

15 The known polymeric dispersants which are based on naphthalenesulfonic acid-formaldehyde condensation products and find use in dye preparations for ink jet printing have the relatively low average molecular weight of about 2 000 to 9 000 g/mol in common.

20 Many of the known dispersants are poorly biodegradable. Readily biodegradable dispersants are the dispersants based on naphthalenesulfonic acid-formaldehyde condensation products that are known from US 5,186,846.

25 Another factor to be borne in mind is the effect of the dispersant on the resulting coloration or print. Many prior art dispersants are not color neutral and taint the printed fabric. Prior art biodegradable naphthalenesulfonic acid-formaldehyde condensation products, for example, distinctly taint printed polyester fabric.

30 It is an object of the present invention to provide dye preparations for printing and especially for ink jet printing which do not have prior art disadvantages. More particularly, the dispersants they contain shall be eliminable from the wastewater, be color neutral in printing and form stable dye dispersions. The dispersants shall be formulatable with the customary assistants or additives.

35 We have found that this object is achieved by dye preparations comprising, each percentage being based on the weight of the preparation,

5 a) from 0.1% to 30% by weight of one or more anthraquinone, quinophthalone or azo dyes which are free of ionic groups as a component (A),
b) from 0.1% to 20% by weight of a dispersant based on a naphthalenesulfonic acid-formaldehyde condensation product having an average molecular weight of at least 11 000 g/mol, as a component (B),
c) from 0.1% to 90% by weight of one or more mono- or polyhydric alcohols as a component (C),
d) from 0% to 5% by weight of customary assistants as a component (D), and
e) if appropriate water ad 100% by weight.

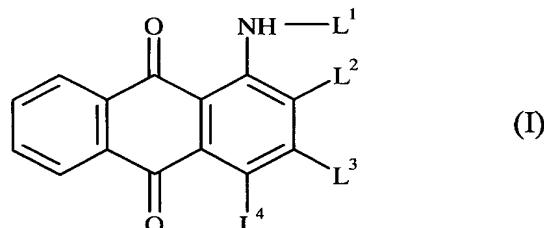
10

The present invention further provides for the use of the dye preparations in ink jet printing processes and other coloration or printing processes.

15

Component (A) of the dye preparations according to the present invention comprises from 0.1% to 30% by weight of one or more anthraquinone, quinophthalone or azo dyes which are free of ionic groups.

20



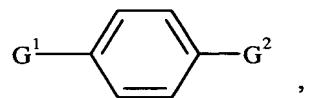
where

25

L^1 is hydrogen, C_1-C_{10} -alkyl or unsubstituted or C_1-C_4 -alkyl-, C_1-C_4 -alkoxy-, halogen- or nitro-substituted phenyl,

L^2 and L^3 are independently hydrogen, unsubstituted or phenyl- or C_1-C_4 -alkylphenyl-substituted C_1-C_{10} -alkoxy, unsubstituted or phenyl-substituted C_1-C_{10} -alkylthio, halogen, hydroxyphenyl, C_1-C_4 -alkoxyphenyl, C_1-C_6 -alkanoyl, C_1-C_6 -alkoxycarbonyl or a radical of the formula

30

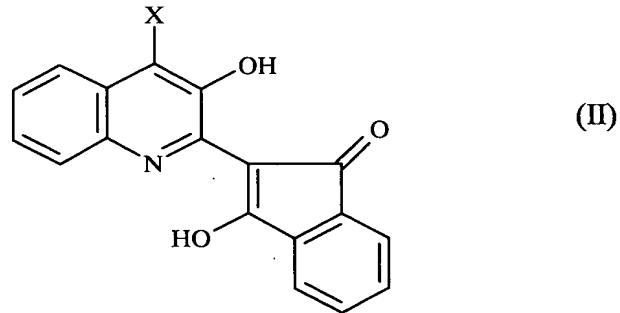


where G^1 is oxygen or sulfur and G^2 is hydrogen or $\text{C}_1\text{--C}_8$ -monoalkylsulfamoyl whose alkyl chain may be interrupted by 1 or 2 oxygen atoms in ether function, and

5

L^4 is unsubstituted or phenyl- or $\text{C}_1\text{--C}_4$ -alkylphenyl-substituted amino, hydroxyl or unsubstituted or phenyl-substituted $\text{C}_1\text{--C}_{10}$ -alkylthio.

Suitable quinophthalone dyes which are free of ionic groups conform for example to
10 formula II



where X is hydrogen, chlorine or bromine.

15

Any alkyl appearing in the abovementioned formula I may be straight-chain or branched.

In any substituted alkyl appearing in the abovementioned formula I the number of substituents is generally 1 or 2.

20

In any substituted phenyl appearing in the abovementioned formulae the number of substituents is generally from 1 to 3 and preferably 1 or 2.

There follows an illustrative exemplification of radicals as defined in the formula I.

25

Alkyl is for example methyl, ethyl, propyl, isopropyl, butyl, isobutyl, sec-butyl, tert-butyl, pentyl, isopentyl, neopentyl, tert-pentyl, hexyl, 2-methylpentyl, heptyl, octyl, 2-ethylhexyl, isoctyl, nonyl, isononyl, decyl or isodecyl (the designations isoctyl,

isononyl and isodecyl are trivial names derived from the oxo process alcohols – cf Ullmann's Encyclopedia of Industrial Chemistry, 5th edition, Vol. A 1, pages 290 to 293, and also Vol. A 10, pages 284 and 285).

5 Phenyl is for example phenyl, 2-, 3- or 4-methylphenyl, 2-, 3- or 4-ethylphenyl, 2-, 3- or 4-propylphenyl, 2-, 3- or 4-isopropylphenyl, 2-, 3- or 4-butylphenyl, 2,3-, 2,4- or 2,6-dimethylphenyl, 2-, 3- or 4-methoxyphenyl, 2-, 3- or 4-ethoxyphenyl, 2,3-, 2,4- or 2,6-dimethoxyphenyl, 2-, 3- or 4-fluorophenyl, 2-, 3- or 4-chlorophenyl, 2-, 3- or 4-bromophenyl or 2-, 3- or 4-nitrophenyl.

10 Alkylthio and phenylthio are each for example methylthio, ethylthio, propylthio, isopropylthio, butylthio, isobutylthio, pentylthio, hexylthio, heptylthio, octylthio, isoocetylthio, 2-ethylhexylthio, nonylthio, isononylthio, decylthio, isodecylthio, benzylthio or 1- or 2-phenylethylthio.

15 Alkoxy is for example methoxy, ethoxy, propoxy, isopropoxy, butoxy, isobutoxy, sec-butoxy, pentyloxy, isopentyloxy, neopentyloxy, tert-pentyloxy, hexyloxy, 2-methylpentyloxy, heptyloxy, octyloxy, isoocetylthio, 2-ethylhexyloxy, nonyloxy, isononyloxy, decyloxy, isodecylthio, benzylthio or 1- or 2-phenylethoxy.

20 Halogen is for example fluorine, chlorine or bromine.

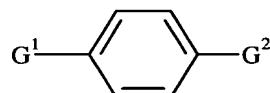
25 Alkoxycarbonyl is for example methoxycarbonyl, ethoxycarbonyl, propoxycarbonyl, isopropoxycarbonyl, butoxycarbonyl, isobutoxycarbonyl, sec-butoxycarbonyl, pentyloxycarbonyl, isopentyloxycarbonyl, neopentyloxycarbonyl or hexyloxycarbonyl.

Alkanoyl is for example formyl, acetyl, propionyl, butyryl, pentanoyl or hexanoyl.

30 Sulfamoyl is for example methylsulfamoyl, ethylsulfamoyl, propylsulfamoyl, isopropylsulfamoyl, butylsulfamoyl, pentylsulfamoyl, hexylsulfamoyl, heptylsulfamoyl, octylsulfamoyl, 2-ethylsulfamoyl, 2-methoxyethylsulfamoyl, 2-ethoxyethylsulfamoyl, 3,6-dioxaheptylsulfamoyl, 3,6-dioxaoctylsulfamoyl, 4,8-dioxanonylsulfamoyl, 3,7-dioxaoctylsulfamoyl, 3,7-dioxanonylsulfamoyl, 4,7-dioxaoctylsulfamoyl, 4,7-dioxanonylsulfamoyl or 4,8-dioxadecylsulfamoyl.

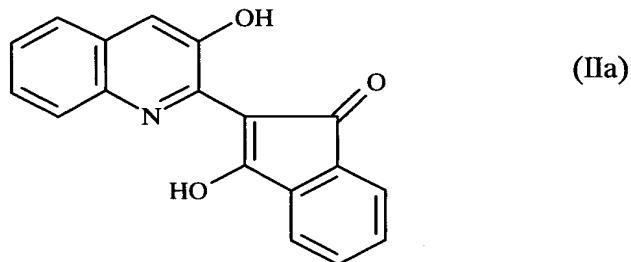
Preference is given to dye preparations comprising one or more anthraquinone dyes of the formula I where L^1 is hydrogen, C_1 – C_4 -alkyl or unsubstituted or methyl-substituted phenyl and L^4 is hydroxyl, amino or unsubstituted or methyl-substituted phenylamino.

5 Preference is further given to dye preparations comprising one or more anthraquinone dyes of the formula I where L^2 is C_1 – C_4 –alkoxy, acetyl, C_1 – C_4 –alkoxycarbonyl or a radical of formula



10 where G^1 and G^2 are each as defined above, with G^1 being in particular oxygen and G^2 in particular hydrogen.

15 Preference is further given to dye preparations comprising the quinophthalone dye of the formula IIa



The dyes of the formulae I and II are generally known dyes. The anthraquinone dyes of the formula I are described for example in K. Venkataraman "The Chemistry of Synthetic Dyes", Vol. III, pages 391 to 413, Academic Press, New York, London, 1970. The quinophthalone dyes of the formula II are described for example in EP-A-83 553 or the literature cited therein.

25 Preference is given to dye preparations wherein 99% of the dye particles are smaller than
1 mm.

Preference is further given to dye preparations comprising dyes of the anthraquinone or quinophthalone series whose sublimation temperature is in the range from 140 to 300°C.

Suitable mono- or polyazo dyes are well known and have been extensively described, for example in K. Venkataraman "The Chemistry of Synthetic Dyes", Vol. VI, Academic Press, New York, London, 1972.

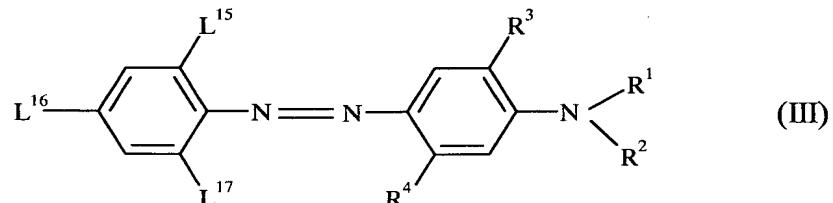
5 Of particular importance are azo dyes, especially monoazo dyes, having a diazo component which is derived from an aniline or from a five-membered aromatic heterocyclic amine which has from one to three heteroatoms selected from the group consisting of nitrogen, oxygen and sulfur in the heterocyclic ring and may be fused with a benzene, thiophene, pyridine or pyrimidine ring.

10 Important monoazo dyes are for example those whose diazo component is derived for example from an aniline or from a heterocyclic amine of the pyrrole, furan, thiophene, pyrazole, imidazole, oxazole, isoxazole, thiazole, isothiazole, triazole, oxadiazole, thiadiazole, benzofuran, benzothiophene, benzimidazole, benzoxazole, benzothiazole, 15 benzisothiazole, pyridothiophene, pyrimidothiophene, thienothiophene or thienothiazole series.

20 Of particular suitability are diazo components derived from an aniline or from a heterocyclic amine of the pyrrole, thiophene, pyrazole, thiazole, isothiazole, triazole, thiadiazole, benzothiophene, benzothiazole, benzisothiazole, pyridothiophene, pyrimidothiophene, thienothiophene or thienothiazole series.

25 Also of importance are azo dyes, especially monoazo dyes, having a coupling component of the aniline, aminonaphthalene, aminothiazole, diaminopyridine or hydroxypyridone series.

Particular preference is given to azo dyes of the formula III



30 where

L^{15} and L^{17} are each cyano,
 L^{16} is C_1-C_6 -alkyl,

R^1 and R^2 are each C_1 - C_6 -alkyl,
 R^3 is hydrogen, C_1 - C_6 -alkyl or C_1 - C_6 -alkoxy, and
 R^4 is hydrogen, C_1 - C_6 -alkyl or C_1 - C_6 -alkanoylamino.

5 As mentioned above, the azo dyes are known per se and described for example in Venkataraman (loc. cit.), in EP-A-201 896, DE-A-3 108 077, US-A-4 843 153, GB-A-1 546 803, EP-A-535 490 or EP-A-544 153 or obtainable by the methods mentioned therein. A list of suitable anthraquinone, quinophthalone or azo dyes can be found for example in the Colour Index not only under disperse dyes but also under vat dyes.

10

Examples of suitable anthraquinone, quinophthalone or azo dyes are

- C.I. Vat Yellow 2, 3, 4, 5, 9, 10, 12, 22, 26, 33, 37, 46, 48, 49 and 50;
- 15 – C.I. Vat Orange 1, 2, 5, 9, 11, 13, 15, 19, 26, 29, 30 and 31;
- C.I. Vat Red 2, 10, 12, 13, 14, 16, 19, 21, 31, 32, 37, 41, 51, 52 and 61;
- C.I. Vat Violet 2, 9, 13, 14, 15, 17 and 21;
- 20 – C.I. Vat Blue 1 (C.I. Pigment Blue 66), 3, 5, 10, 12, 13, 14, 16, 17, 18, 19, 20, 22, 25, 26, 29, 30, 31, 35, 41, 42, 43, 64, 65, 66, 72 and 74;
- C.I. Vat Green 1, 2, 3, 5, 7, 8, 9, 13, 14, 17, 26, 29, 30, 31, 32, 33, 40, 42, 43, 44 and 49;
- 25 – C.I. Vat Brown 1, 3, 4, 5, 6, 9, 11, 17, 25, 32, 33, 35, 38, 39, 41, 42, 44, 45, 49, 50, 55, 57, 68, 72, 73, 80, 81, 82, 83 and 84;
- 30 – C.I. Vat Black 1, 2, 7, 8, 9, 13, 14, 16, 19, 20, 22, 25, 27, 28, 29, 30, 31, 32, 34, 36, 56, 57, 58, 63, 64 and 65;

35 Component (B) of the pigment preparations according to the present invention comprises from 0.1 to 20% by weight of a water-soluble dispersant based on a naphthalenesulfonic acid-formaldehyde concentration product having an average molecular weight of at least 11 000 g/mol.

The naphthalenesulfonic acid-formaldehyde condensation products used according to the present invention as dispersants preferably have an average molecular weight of from 11 000 to 35 000 and more preferably from 11 000 to 20 000 g/mol.

5 In general, the fraction having a molecular weight above 11 000 g/mol is in the range from at least 5% to not more than 95% and preferably in the range from 10% to 90%.

Preferred dispersants (B) are condensation products having a sulfonic acid group content of not more than 40% by weight.

10 The condensation products (B) are obtainable by sulfonation of naphthalene and subsequent condensation of the resultant naphthalenesulfonic acids with formaldehyde.

15 The naphthalenesulfonic acid-formaldehyde condensation products used according to the present invention are preferably prepared in accordance with the following general method of making:

20 1–3 parts by weight of naphthalene are sulfonated with 1–3 parts by weight of a sulfuric acid having a concentration of 85–100% by weight or oleum having a free SO₃ content of from 2% to 45% by weight. The sulfonation can be carried out at from 80 to 190°C, the reaction times being from 0.5 to 10 hours. The sulfonation can be carried out in the presence of assistants such as boric acid, preferably in a concentration of from 0.5% to 5% by weight, based on sulfuric acid or oleum. After the sulfonation, the reaction mixture is diluted with from 0.5 to 2 parts of water and subsequently condensed with from 0.3 to 25 1.8 parts of aqueous formaldehyde solution having a concentration of from 20% to 40% by weight of formaldehyde at from 80 to 180°C. The condensation mixture is subsequently diluted with up to 0.5 part of water and adjusted with aqueous sodium hydroxide solution to a pH in the range from 4 to 10. The condensation mixture is finally diluted once more with 0.5 part of water, admixed with aqueous sodium hydroxide solution and milk of lime 30 and filtered to remove precipitated CaSO₄. The pH is subsequently adjusted to a value in the range from 4 to 10. Finally, water is used to set the final concentration in the range from 15 to 50% by weight of dry content.

35 This method of making gives a naphthalenesulfonic acid-formaldehyde condensation product having the desired molecular weight.

Component (C) of the dye preparations according to the present invention comprises from 0.1% to 90% by weight of one or more mono- or polyhydric alcohols.

5 Suitable mono- or polyhydric alcohols are generally alkanemonoools or -polyols, especially polyols having from 2 to 8 carbon atoms, preferably from 2 to 6 carbon atoms and up to 4 and preferably from 2 to 4 alcoholic hydroxyl groups. Specific examples are 1,2-ethanediol, 1,2-propanediol, 1,3-propanediol, glycerol, 1,2,5-pantanetriol or 1,2,6-hexanetriol, 1,2-hexanediol and 1,2-pantanediol.

10 The dye preparations may further comprise, based on the weight of the preparation, up to 10% by weight and preferably from 0.1% to 10% by weight of a polyalkylene glycol.

15 Suitable polyalkylene glycols for inclusion in the dye preparations according to the present invention are in particular polyethylene or polypropylene glycols which have for example an average molecular weight in the range from 100 to 1 000 and preferably in the range from 100 to 600 and especially about 400. If desired, ethylene oxide-propylene oxide copolymers can also be used.

20 Preference is given to dye preparations which, each percentage being based on the weight of the preparation, comprise from 1% to 50% by weight of one or more anthraquinone, quinophthalone or azo dyes as component (A), from 0.5% to 20% by weight of dispersant (B) and from 1% to 80% by weight and preferably from 2% to 50% by weight of one or more mono- or polyhydric alcohols as component (C).

25 The dye preparations according to the present invention may comprise customary assistants, such as preservatives, antioxidants, foam preventatives, surfactants or viscosity regulators, as a component (D). These agents are known per se and commercially available. When these agents are present in the dye preparations according to the present invention, their total amount will be up to 5% by weight and preferably up to 1% by weight, based on the weight of the preparation.

30 Preferred dye preparations comprise surfactants to reduce the surface tension and to improve the wettability in the ink head.

35 Preferred dye preparations comprise surfactants based on ethoxylated or propoxylated fatty or oxo process alcohols, propylene oxide-ethylene oxide block copolymers, ethoxylates of

oleic acid or alkylphenols, alkylphenol ether sulfates, alkylpolyglycosides, alkyl phosphonates, alkylphenyl phosphonates, alkylphenyl phosphates or alkynediols.

The sum total of the constituents in the dye preparations according to the present invention is obviously 100% by weight in each case. When the sum total of the constituents of the dye preparations according to the present invention is a value less than 100% by weight, the remainder will generally be water (component E).

The water content of dye preparations according to the present invention which are used as printing inks is typically in the range from 50% to 90% by weight, based on the total weight of the dye preparations.

The surface tension of the dye preparations according to the present invention is generally in the range from 20 to 70 Nm/m and preferably in the range from 25 to 60 Nm/m.

The viscosities of the dye preparations according to the present invention is generally in the range from 2 to 300 mPa·s and preferably in the range from 2 to 150 mPa·s.

The pH of the dye preparations according to the present invention is generally in the range from 5 to 11 and preferably in the range from 6 to 10.

The novel dye preparations are prepared in a conventional manner. For instance, the dye, for example in the form of a press cake, can be mixed together with the dispersant, the mono- or polyhydric alcohol and optionally polyalkylene glycol in the presence of water and predispersed in a suitable apparatus. The resulting mixture can then be treated in a mill to achieve the desired dye particle size. Finally, the final adjustment can be effected by adding appropriate amounts of water, optionally polyalkylene glycol and optionally further assistants and, after mixing, filtration by means of a sieve, preferably a sieve having a pore size of 1 mm.

The dye preparations according to the present invention are very useful as inks in the ink jet process and also for sublimation transfer printing.

The ink jet printing process is usually carried out with aqueous inks, which are sprayed as small droplets directly onto the substrate. There is a continuous form of the process, in which the ink is pressed at a uniform rate through a nozzle and the jet is directed onto the substrate by an electric field depending on the pattern to be printed, and there is an

interrupted ink jet or drop-on-demand process, in which the ink is expelled only where a colored dot is to appear, the latter form of the process employing either a piezoelectric crystal or a heated hollow needle (bubble or thermal jet process) to exert pressure on the ink system and so eject an ink droplet. These techniques are described in Text. Chem. 5 Color, Volume 19 (8), pages 23 to 29, 1987, and Volume 21 (6), pages 27 to 32, 1989.

The dye preparations according to the present invention are particularly useful as inks for the bubble jet process or for the process employing a piezoelectric crystal.

10 Useful substrates for the ink jet process include not only paper but also the hereinbelow recited substrate materials.

15 Suitable substrates are in particular textile materials, for example fibers, yarns, threads, knits, woven or non-woven composed of polyester, modified polyester, for example anionically modified polyester, blend fabrics of polyester with cellulose, cotton, viscose or wool, polyamide, polyacrylonitrile, triacetate, acetate, polycarbonate, polypropylene or polyvinyl chloride, polyester microfibers or else polymer-coated substrates, such as metal foils, glass or ceramic.

20 The dye preparations according to the present invention are particularly useful for printing onto textile. The substrate, which depends on the class of the dye (vat dye, disperse dye or a mixture thereof), is cotton, viscose, polyester or a blend fabric of cotton or viscose and polyester. The print can be effected directly or indirectly by sublimation transfer printing.

25 In sublimation transfer printing, a pattern is initially preformed on a transfer and then transferred by means of heat to a substrate. The dye can be fixed not only in the course of the transfer process itself but also in a subsequent fixation and after-treatment operation. This process is common knowledge and described for example in Ullmann's Encyclopedia of Industrial Chemistry, 5th Edition, Volume A26, pages 499 to 501.

30 The novel dye preparations are notable for not causing ink jet nozzle blockages. Furthermore, their use leads to stripe-free prints.

The examples which follow illustrate the invention.

Examples

Dispersants 1, 2 and 3 are commercial dispersants of the kind obtainable on the market.

Dispersant 1 is a naphthalenesulfonic acid-formaldehyde condensation product which has an average molecular weight of about 6 000. Dispersant 2 is an aqueous solution of a

5 naphthalenesulfonic acid-formaldehyde condensation product which likewise has an average molecular weight of about 6 000. Dispersant 3, by contrast, is a liquid formulation of an arylsulfonic acid-formaldehyde condensation product which has an average molecular weight of about 9 000 g/mol.

10 Dispersant 4 used according to the present invention is a liquid formulation of naphthalenesulfonic acid-formaldehyde condensation product having an average molecular weight of 16 000 and an active content of 37%. The dispersant 5 according to the present invention is a pulverulent formulation having an average molecular weight of 18 000 g/mol and an active content of 79%. In addition, dispersants 4 and 5 are more than 90%

15 eliminable from wastewater.

Inventive examples 1 to 4 and comparative examples 1 to 5

Dispersing properties of dispersants

20 The dispersing properties were determined by means of dispersing tests and grinding tests.

The dispersing properties of dispersants for disperse dyes were determined by the following method:

25 100 ml of a dispersion from 0.5 g of Palanil Scarlet and x g of dispersant are adjusted to pH 5 and maintained at 130°C under the autogenous pressure for 30 min. This is followed by cooling to 63°C and filtration of the resulting dispersion through a blue ribbon filter at 63°C. The observable residue is subsequently visually evaluated.

30

35

Example	Component	Amount used/ effect		Amount used/ effect		Amount used/ effect	
		g/l	g/l	g/l	g/l	g/l	g/l
comp 1	dispersant 1			1	moderate	2	good
comp 2	dispersant 2			2	moderate	4	good
comp 3	dispersant 3			2	moderate	4	good
inv 1	dispersant 4			2	good	4	very good
inv 2	dispersant 5	0.5	moderate	1	good	2	very good

It is found that dispersants 4 and 5 used according to the present invention have distinctly better dispersing characteristics than the commercial comparative products.

5 The dispersing properties of dispersants for vat dyes were determined by the following method:

100 ml of stock vat consisting of 0.5 g/l of Indanthren Brilliant Violet RRN, 12 ml/l of aqueous sodium hydroxide solution (38°Bé) and 4 g/l of sodium dithionite (100%) are then
 10 mixed with x g of dispersant. This is followed by addition of 5 ml of 6% by weight of hydrogen peroxide solution with stirring and stirring for about 1 min. A white ribbon filter from Schleicher & Schüll (90 mm in diameter, No. 300109) is placed onto the open side of a beaker and 1 ml of the liquor obtained is pipetted into the center of the filter. The filter is then air dried before the spot test is evaluated by visual inspection. Poor dispersants exhibit
 15 distinct agglomerations, whereas good dispersants exhibit a distinct flow of finely dispersed, reoxidized vat pigments across the filter paper.

Example	Component	Amount used/ effect		Amount used/ effect		Amount used/ effect	
		g/l	g/l	g/l	g/l	g/l	g/l
comp 4	dispersant 1	0.5	moderate	1	good	2	good
comp 5	dispersant 2	1	moderate	2	good	4	very good
inv 3	dispersant 4	0.5	moderate	1	good	2	very good
inv 4	dispersant 5	0.5	moderate	1	good	2	very good

It is again clear that dispersants 4 and 5 used according to the present invention have
 20 distinctly superior dispersing properties.

Example 5

Evaluation of tainting by dispersants

5 g of polyester fabric were dyed for 60 min at 130°C in a liquor ratio of 20:1 in a blank dyeing liquor containing 5 g/l of the dispersant to be tested and acetic acid buffer to adjust the pH to 4.5. This is followed by rinsing with demineralized water before the textile was dried and visually inspected to evaluate tainting.

Assistant	Evaluation
dispersant 1	good
dispersant 2	good
dispersant 3	poor
dispersant 4	good
dispersant 5	good

10 Dispersants 4 and 5 used according to the present invention are at least equivalent to the prior art dispersants with regard to tainting.

Example 6

Grindability of dispersant 4 compared with dispersant 3

15 15 g of Disperse Red 60, 7.5 g of polyethylene glycol (average molecular weight 400 g/mol), 15 g of dispersant 3 or dispersant 4, 0.4 g of a 50% by weight solution of glutaraldehyde and 0.5 g of a 47% by weight solution of tetramethylolacetylenediurea are bulked with water to a total weight of 100 g and pasted up in a mill. Thereafter, the pH is
20 adjusted to 8.5 with 0.5 part of triethanolamine and subsequently the mixture is ground in a stirred media mill. The particle size following a defined energy input by the mill is determined and compared.

kWh/t	Dispersant 3		Dispersant 4	
	x50	x16	x50	x16
2000	0.52	1.00		
3000	0.44	0.87	0.37	0.74
4000	0.39	0.77	0.35	0.69
5000	0.36	0.71	0.33	0.66

Here, $\times 50$ and $\times 16$ mean that respectively 50% and 16% of the particles have a size above the stated value. The above table reveals the grinding progress. It is clear that dispersant 4 gives a distinctly better grinding result than dispersant 3.

5

Example 7

Preparation and testing of mixing component

In a dissolver, 15.0 g of Disperse Red 60, 6.13 g of polyethylene glycol (average molecular weight 400 g/mol), 33.0 g of dispersant 4 used according to the present invention, 0.4 g of a 50% aqueous solution of glutaraldehyde, 0.5 g of a 4% by weight aqueous solution of tetramethylolacetylenediurea and 0.5 g of triethanolamine were mixed and made up with 35.56 g of demineralized water. This mixture was ground in a ball mill from Molynex for about 5 hours until the maximum particle diameter was $1\text{ }\mu\text{m}$. 10 g of the mixing component was heated at 60°C for 14 days, during which no signs were observed of disperse dye separating from the dispersion or of particle growth.

Example 8

Making of an ink

20

In a beaker, 40 g of the particular mixing component were mixed with 30 g of glycerol, 10 g of polyethylene glycol (average molecular weight 400 g/mol), 0.4 g of heptamethyltrisiloxane allyl alkoxylate copolymer and 119.6 g of demineralized water. The ink thus obtained was printed by an EPSON 3000 Stylus Color piezo head printer onto A3 paper. The prints obtained had excellent line crispness. There was no nozzle failure after 40 sheets of paper, nor after 10 m^2 of printing in sustained use. The flow properties of the ink are excellent. The printing tests were repeated after the ink had been aged at 60°C for 5 days; again, a satisfactory print and flow was observed. After aging of the ink at 60°C for 21 days, no separation or disperse dye from the dispersion and no particle growth were observed. The surface tension of the ink was 34 mN/m and the viscosity 3.6 mPa s.

Similar results are obtained for example with inks based on Disperse Blue 72, Disperse Blue 332, Disperse Blue 359, Disperse Yellow 54, Disperse Blue 60, Disperse Blue 77, Disperse Blue 73, Disperse Red 86, Disperse Red 91, Disperse Red 92, Solvent Yellow 163, Disperse Red 277, Disperse Orange 44, Disperse Yellow 198, Disperse Red 279, Vat Red 41, Vat Blue 1, Vat Blue 5, Vat Yellow 46.